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IS 15081 (2001): N, N-Bishydroxyethyl Metachloroaniline, Technical [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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Indian Standard

N, N-BISHYDROXYETHYL META-CHLOROANILINE, TECHNICAL — SPECIFICATION

ICS 71.080.80

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

December 2001

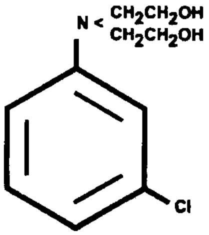
Price Group 3

Dyes Intermediate Sectional Committee, PCD 11

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Dyes Intermediate Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

N,N-Bishydroxyethyl meta-Chloroaniline ($C_{10}H_{14}O_2NCl$) is an important dye intermediate used in the manufacture of azo disperse dyes. It has the following structural formula:



N,N-Bishydroxyethyl meta-Chloroaniline
[(Molecular Mass 215.5)
CAS Registry No.(92-00-2)]

The Composition of the Committee responsible for formulation of this standard is given in Annex B.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

N, N-BISHYDROXYETHYL META-CHLOROANILINE, TECHNICAL — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and the methods of sampling and tests for N,N-Bishydroxyethyl meta-Chloroaniline.

2 REFERENCES

The following standards contain provisions which through reference in this text constitute provisions of this standards. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
2552 : 1989	Steel drums (galvanized and ungalvanized) (*third revision*)
5299 : 2001	Methods of sampling and tests for dye intermediates (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of cream solid material.

3.2 The material shall also comply with the requirements given in Table 1.

Table 1 Requirements for N,N-Bishydroxyethyl meta-Chloroaniline

Sl No.	Characteristic	Requirement	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Melting range, °C	87-90	9 of IS 5299
ii)	Assay, percent by mass, *Min*	97.0	A-2 & A-4
iii)	N-hydroxyethyl meta-Chloroaniline, *Max*	1.5	A-4
iv)	meta-Chloroaniline (on dry basis), *Max*	0.2	A-3 & A-4
v)	Insoluble in methanol, percent by mass, *Max*	0.3	11 of IS 5299

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in steel drums (*see* IS 2552) lined with suitable polyethylene film or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

4.2 Marking

Each container shall bear legibly and indelibly the following information:

a) Name of the material,
b) Indication of the source of manufacture,
c) Net mass of the material,
d) Batch number, and
e) Month and year of the manufacture.

4.3 BIS Certification Marking

The container may also be marked with the Standard Mark.

4.3.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in **4** of IS 5299.

5.2 Number of Tests

5.2.1 Tests for the determination of N,N-Bishydroxyethyl meta-Chloroaniline, meta-Chloroaniline and N-hydroxyethyl meta - Chloroaniline shall be conducted on each of the individual samples.

5.2.2 Tests for the determination of remaining characteristics, namely, description, melting range and solubility shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 *For Individual Samples*

The lot shall be declared as conforming to the requirement of N,N-Bishydroxyethyl meta-Chloroaniline, meta-Chloroaniline and N-hydroxyethyl meta-Chloroaniline, if each of the individual test results satisfies the relevant requirements given in Table 1.

5.3.2 *For Composite Sample*

For declaring the conformity of the lot to the requirements of all other characteristics (*see* **5.2.2**) tested on the composite sample, the test results for each of the characteristics shall satisfy the relevant requirements given in **3.1** and Table 1.

6 TEST METHODS

6.1 Tests shall be carried out according to the methods refered in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Sl No.*(ii),(iii) *and* (iv)]

DETERMINATION OF N,N-BISHYDROXYETHYL META - CHLOROANILINE AND ITS IMPURITIES

A-1 GENERAL

Two methods, namely, 'Titration Method' (Method A-2) and Gas Liquid Chromatography (GLC) (Method A-4) have been prescribed for the determination of N, N-Bishydroxyethyl meta-Chloroaniline and its impurities. Any of these two methods can be used on routine basis. However in the event of dispute, GLC method shall be treated as referee method. Reference may be made to IS 5299 for details of GC test method to be followed. However, necessary details of test conditions are given here for guidance only.

A-2 TITRATION METHOD

A-2.1 Outline of the Method

A diazonium solution is prepared by reacting 2,4 Dinitroaniline in concentrated sulphuric acid with sodium nitrite solution. It is standardized by titrating against N, N-Bishydroxyethyl meta-Chloroaniline zof known purity. The sample of N, N-Bishydroxyethyl meta-Chloroaniline is then titrated against this standard diazonium solution.

A-2.1.1 *Reagents*

A-2.1.2 *Glacial Acetic Acid*

A-2.1.3 *Dilute Sulphuric Acid* (*20 percent m/v*)

A-2.1.4 *Dilute Acetic Acid (20 percent m/v)*

A-2.1.5 *Sodium Acetate Solution (20 percent m/v)*

A-2.1.6 *H-Acid Indicator Solution* — Dissolve 0.5 g of H-acid in 100 ml of water containing 1 g of soda ash.

A-2.1.7 2,4 *Dinitroaniline* 99 *Percent Pure*

A-2.1.8 *N, N-Bishydroxyethyl meta-Chloroaniline* — Reference sample of known purity.

A-2.2 Procedure

A-2.2.1 *Preparation of* 2,4 *Dinitroaniline Diazo Solution*

Take 600 g (326 ml) of concentrated H_2SO_4 in a reaction vessel with stirrer which is cooled to 10°C externally on ice bath. Add 69 g of (100 percent) sodium nitrite gradually under stirring maintaining the temperature below 10°C. After the addition is complete, remove the ice bath and bring it to room temperature and then raise to 60°C to dissolve the material completely. After the dissolution is complete the temperature is again brought below 20°C. Similarly, prepare a slurry of 2, 4 Dinitroaniline in H_2SO_4 by dissolving 183 g of 2, 4 Dinitroaniline in 200 g of H_2SO_4 in a 500 ml beaker. Add this slurry to the nitrosulphuric acid slurry prepared earlier maintaining the temperature below 20°C. After the addition is complete, stir for another 2 h for the completion of reaction (stock solution). Depending upon the requirements, a portion of this slurry is taken out and diluted with 10 times by its volume with ice water (for exmaple, 10 ml of stock solution and 90 ml of ice water). The solution is then clarified with activated carbon and filtered before use. It is to be noted that the diluted diazo solution is not stable and hence should be prepared fresh every time before use.

A-2.2.2 *Standardization of* 2,4 *Dinitroaniline Diazonium Solution*

Weigh exactly 0.780 8 g of N, N-Bishydroxyethyl meta-Chloroaniline (99 percent) and transfer it to a 1 litre beaker. Dissolve it with 10 - 15 ml of glacial acetic acid and dilute the solution with ice water to 500 ml. Add about 100 ml of 20 percent sodium acetate solution (*m/v*) to bring the *p*H between 4 to 6. Titrate with 2,4 Dinitroaniline diazo (diluted) until the solution shows the presence of an excess diazo with H-acid (bluish black coloration). This may be checked by taking a drop of the reaction mixture and a drop of a diluted solution of H-acid on a piece of filter paper. The bluish black coloration in the junction of the two liquids indicates that the end point has reached. Throughout the titration, the *p*H should be maintained between 4 to 6.

A-2.2.3 Repeat the titration as presented in **A-2.2.2** using the test sample of N, N-Bishydroxyethyl meta-Chloroaniline.

A-2.3 Calculation

A-2.3.1 Normality of diazonium solution

$$N=\frac{P\times 0.780}{V\times 21.55}$$

where

P = purity (percent by mass) of the known sample, and

V = volume in ml of diazonium solution required for the known sample.

A-2.3.2 Assay, percent by mass $=\frac{V_1\times N\times 21.55}{0.780}$

where

V_1 = volume in ml of diazonium solution required for the test sample, and

N = normality of diazonium solution.

A-3 DETERMINATION OF META-CHLOROANILINE BY TLC

A-3.0 Outline of the Method

Meta-Chloroaniline is determined by thin layer chromatography. Reference may be made to IS 5299 for details of TLC test method to be followed. However, necessary details of test conditions are given here for guidance only.

A-3.1 Apparatus

A-3.1.1 Thin layer chromatographic chamber 25 cm × 25 cm.

A-3.1.2 Micropipette of 10 microlitre capacity.

A-3.1.3 Thin layer chromatography plates size 20 cm × 20 cm.

A-3.2 Reagents

A-3.2.1 *Meta-Chloroaniline Pure* — (reference sample). Prepare 0.001 percent,0.002 percent and 0.004 percent, solution in methanol.

A-3.2.2 Eluent Benzene : Ethylacetate : Glacial acetic acid (60 : 39 : 1) *v/v*.

A-3.2.3 Nitrous Fumes Chamber

A glass chamber of size 25 cm × 25 cm with a beaker of capacity 100 ml containing approximately. 50 ml of concentrated HCl (30 percent *m/v*). The nitrous fumes are generated by adding a little solid sodium nitrite everytime before inserting the plate.

A-3.2.4 Spray reagent 0.5 percent solution of American Base (N-1-Naphthylethylene diamine dihydrochloride) in Methanol (*w/v*).

A-3.3 Procedure

Weigh 2 g of the sample under test (100 percent basis) and dissolve in 100 ml methanol. Take a plate coated with silica gel *G* and spot 10 ml of each of the test solution and reference solution along the start line a distance of 3 cm from one another. Put the eluent and insert the plate immediately in the chamber without saturating the chamber and allow to run for about 25-30 min. During this time the solvent from travels approximately 15 cm past the start line. Remove the plate from the chamber, dry it in air. After drying put the plate in the nitrous fumes by blowing air on it. Now spray the plate with American base when the spots (red violet) characterisitc of meta-Chloroaniline become visible at a Rf value of 0.8.

A-3.4 Reporting

Estimate the quantities of the impurity by comparing the colour intensity of the spots of the material under test with those of the solutions of the reference sample.

A-4 DETERMINATION OF ASSAY AND IMPURITIES BY GLC

A-4.0 Outline of the Method

The contents of N,N-Bishydroxyethyl meta-Chloroaniline and possible impurities, namely, N-hydroxyethyl meta-Chloroaniline and meta-Chloroaniline are determined by gas liquid chromatographic method. For details of test method, *see* IS 5299.

A-4.1 Apparatus

Any gas chromatograph capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks may be used. A typical chromatogram obtained with Perkin Elmer instrument under the following conditions is shown in Fig. 1:

Column	
Material	Stainless steel
Length	2 000 mm
Internal diameter	2.75 mm
Stationary phase	10 percent OV 1
Solid Support	on chromosorb W/HP mesh 80/100
Carrier Gas	Nitrogen
Flow rate	30 ml/min
Condition	Oven temperature a) initial temperature 100 °C for 2 min. b) final temperature 200 °C with a programming rate of 10°/min
Injection Temperature	325 °C
Detector Temperature	325 °C

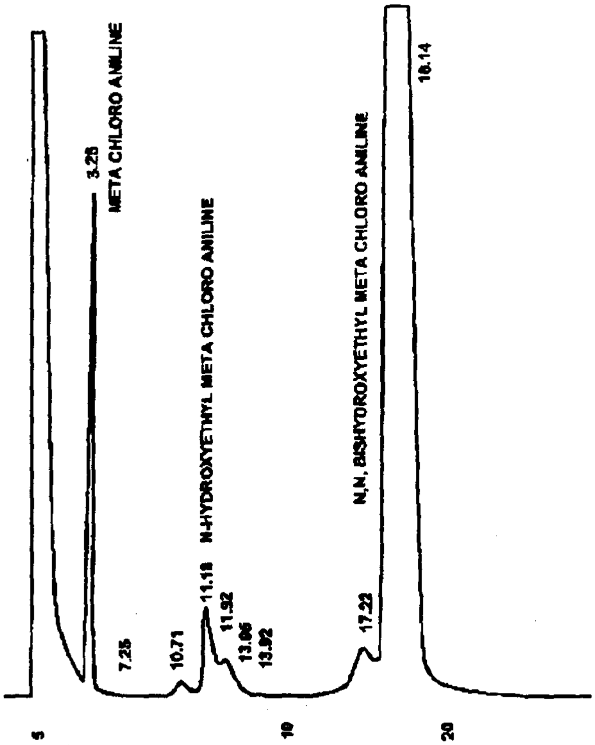


FIG. 1 A TYPICAL CHROMATOGRAM OF N,N- BISHYDROXYETHYL META-CHLOROANILINE

Detector	Flame ionization
Chart speed	5 mm/min.
Sample size	0.5 microlitre
Retention time	
meta-Chloroaniline	3.25 min
N-hydroxyethyl meta-Chloroaniline	10.7 min
N,N-Bishydroxyethyl meta-Chloroaniline	17.22 min

A-4.2 Interpretation of Chromatogram

The peaks for of N,N-Bishydroxyethyl meta-Chloroaniline and possible impurities, namely, N-hydroxyethyl meta-Chloroaniline and meta-Chloroaniline get separated. A typical Chromatogram under the conditions given above is shown in Fig. 1.

A-4.3 Calculation

Follow the procedure given below for calculation when internal standard method is employed:

Percent by mass of N,N-Bishydroxyethyl meta-Chloroaniline =

$$\frac{\text{Concentration of standard} \times \text{Area of peak in the sample} \times \text{Strength of standard}}{\text{Concentration of sample} \times \text{Area of peak of standard}}$$

Similarly other fractions can also be calculated.

ANNEX B

(*Foreword*)

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Dyes Intermediate Sectional Committee, PCD 11

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